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A Review of Pulsed Plasma Synthesis Methods for Preparation of Polymer Thin Films

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ABSTRACT

Conducting polymers are prominent materials in respect of their easy synthesis in the form of thin films and sensitive response regarding their physical and chemical properties. We in this paper present an overview of different pulsed plasma polymerization methods for synthesis of polymer films tunable catechol bearing thin films and plasma coatings from acetylene, acrylic acid and maleic anhydride plasma. After going through over 40 recent works of plasma polymerization methods using pulsed plasma technique, we have discussed the existing challenges and have suggested the ways which can help students and researchers intended to work in polymerization field with simple and useful plasma synthesis methods for preparation of polymer thin films.

KEYWORDS: *Conducting polymer, Polymerization, Pulsed plasma, Polymer films*

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INTRODUCTION

A new path in the sensing applications and detection of hazardous gases in the environment is being explored with the application of conducting polymer based sensors. Conducting polymer synthesized in the form of thin films have been used in sensing applications. Many review papers and studies in previous years have been presented using plasma polymerization techniques. However very few of them provide the convincing view of plasma polymerization techniques for aniline thin films.

Good conductivity, greater stability, higher solubility in organic solvents ease of preparation of various conducting polymers such as polpyrrole, polystyrene, polyphenylene and polyaniline are the benefits that make conducting polymers attractive.

- In the other way with the knowledge of versatile chemistry of polyphenols and catechols. The catechols based polymers are reported which are valuable in production of water-resistant and biomedical adhesives.
- In another method, thin film coatings from maleic anhydride plasma acetylene and acrylic acid are taken as functional groups in the plasma adhesive bond.
- In all above synthesis methods pulsed plasma polymerization technique is used with different suitable methodologies.

One of the first documented studies by N. Bondt et.al. is 1790s reports for polymer synthesis methods as the first document de wilde et.al. [1], Berthelot et.al. [2,3] studied arc synthesis of hydrocarbons. German scientist presented the first study of synthesis of various organics using glow discharge [4-7].

Goodman [8] reported the first application of these plasma polymers. Further studies reported applications [9-14] of various substances with property improvement of materials. Plasma synthesis is now adopted for various application such as surface modification [15-18] and layer deposition for electrical devices [19-22]. Plasma state is ionized

gases consisting of photons, electrons, ions, neutral particles, radicals and metastable particles. Plasma is free space with equal number of positive and negative charged particles behave as neutral [23,24].

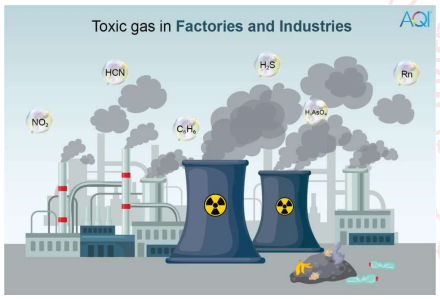
These plasma can artificially be generated in the laboratory by electric discharge [25,26]. Plasma generated by this method is called non thermal or cool plasma [27]. This technique provides a safeguard to substrates or substances due to low temperature of heavy particles. Plasma polymerization is advantageous due to its simple installation, fast processing, low cost, low temperature and green synthesis [28-31].

In this paper, we have presented plasma polymerization with pulsed plasma technique by different approaches and have compared plasma parameters through characterization of plasma polymers, like Fourier Transform Infrared spectroscopy (FTIR), X-ray Photoelectron Spectroscopy (XPS). Also through kinetics of deposition and dependence of chemical and physical properties. Our purpose in this review is to provide a reference to recent devices and methods of pulsed plasma polymerization and hope that student and researchers working to synthesize plasma polymers can select the best method for their experiments.

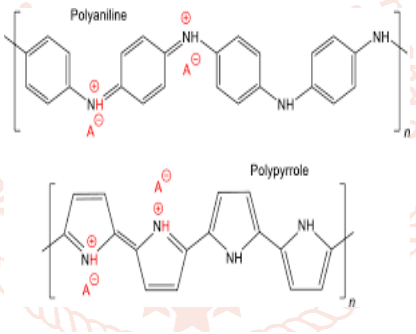
Polyaniline (PANI) is the best sensing material among conducting polymers. The response conditions determine their morphology, structure and physical properties. The semiconducting properties of PANI thin films modulated by the absorption and de-absorption of gases depicted significant role in chemical sensors [32]. Pulsed plasma method has been widely use for the preparation of semiconducting polymer films mainly (PANI) [33]. The film so prepared was analyzed for its electrical, optical, and structural characteristics. Two main drawbacks of plasma polymers are

1. They have poor selectivity and easily react with chemical agent.
2. Even after accurate measurements; cause the sensor signal to be non-linear.

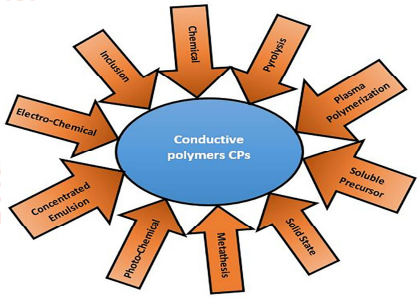
Today sensors are very much used for environmental monitoring and protection [35]. These sensors are fabricated with the polymer thin films, which are polymerized by plasma methods with which we are mainly concerned in our present study.



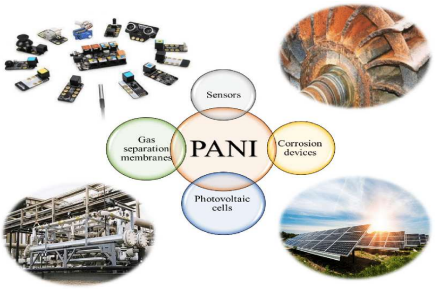
1. Toxic gases in factories and industries.



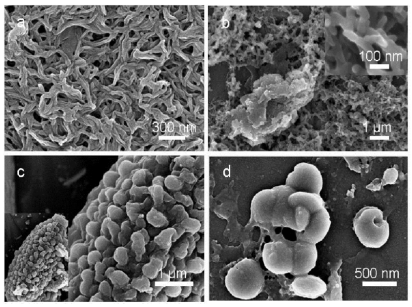
(b) Polyaniline formation



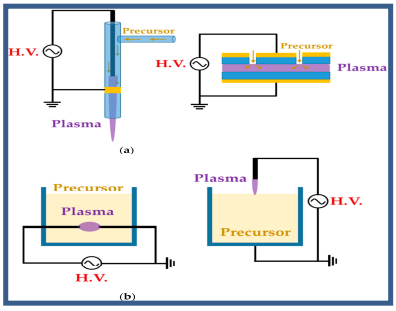
(c) Conducting polymer



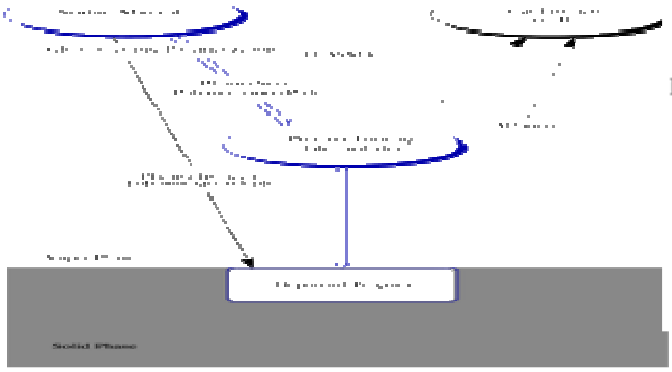
(d) Applications of PANI



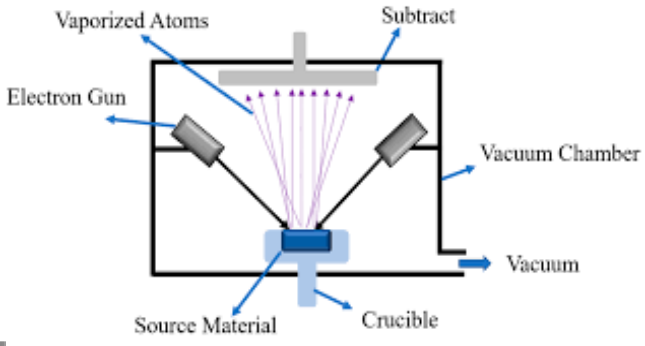
(e) PANI morphology



(f) Plasma polymerization is a process of synthesis thin polymeric film in a low-pressure



(g) Plasma polymerization technique



(h) On coating techniques for surface protection

In recent years polymer has attracted people's attention for its new invention. Being light strong and flexible and suitability of thermal, electrical, high specificity, and oxidation resistance, these are very useful for optoelectronics, biosensors, chemical sensors, and electronic display. Plasma polymerization is a process of synthesizing polymeric thin films in low pressure electric discharge [37]. This technique is being applied for protection of membranes for reverse osmosis, insulating layers for semiconductors and protective coatings for optical components, we have focused too.

Here we present a review on pulsed plasma deposition techniques, materials, and their characterization by different workers with an inclusion of their basic instrumentation.

Experimental:

Materials and methods:-

We have studied the experimental arrangements of different workers for plasma polymerization.

Inductively coupled Pulsed Plasma Polymerization (PPP):-

Rajendra Kumar et.al. (38) used pulsed plasma polymerization to make a variety of films. Using conducting polymer such as polyaniline [39, 40, 41], polyethylene [42], Polypyrrole [43] etc. In their method a number of volatile organic compounds could be use to the plasma reactor. In their system contamination of the film could be avoided due to no use of oxidants and solvents [44]. A thermocouple gauge is used with capacitance monometer to monitor reactor pressure. Thermocouple guage is calibrated in absence of plasma and during deposition of film it was removed. After enough monomer has reached to reactor, 1.8 µF capacitor was discharged and during activation of plasma, with decay time constant 10µs, the RF coil was excited with a damped sinusoid of 290kHz. After completing 10 such plasma shots the reactor was evacuated and fresh monomer vapor was refilled. The substrate holder and RF coil separations were varied. Glass plates and indium tin oxide (ITO) were used as substrates and the choice of substrate depended on the method of characterization. The reagent-grade aniline (Aldrich; 98.5) monomer purified with triple distillation were stored in a dark bottle in nitrogen affinity. Profilometer was used to measure thickness of plasma polymerized aniline films.

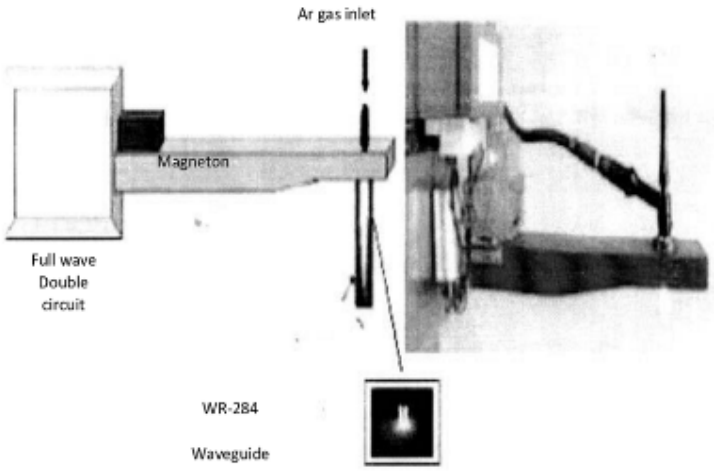


Fig. 1. Shows a typical schematic diagram of plasma polymerization unit with an argonatmospheric plasma brush

Atmospheric Aerosol Assisted (PPP):-

In another working Vincent Jalaber et.al. [45], who used an environmentally friendly technique of pulsed plasma technique where plasma deposition were performed on mirror polished stainless steel (ss) disks and slicon wafers polished on both faces. A home synthesized Dopamaine Acrylamide (DoA) was produced according as Patil et. al. (2015) [46]

The 2-hydroxyethyl methacrylate monomer (HEMA 97%), the micro-BCA protein assay kit, liquid precursor mixture were managed in their desired form. The substrates were cleaned with acetone and absolute ethenol for 5minutes each and dried under nitrogen flux.

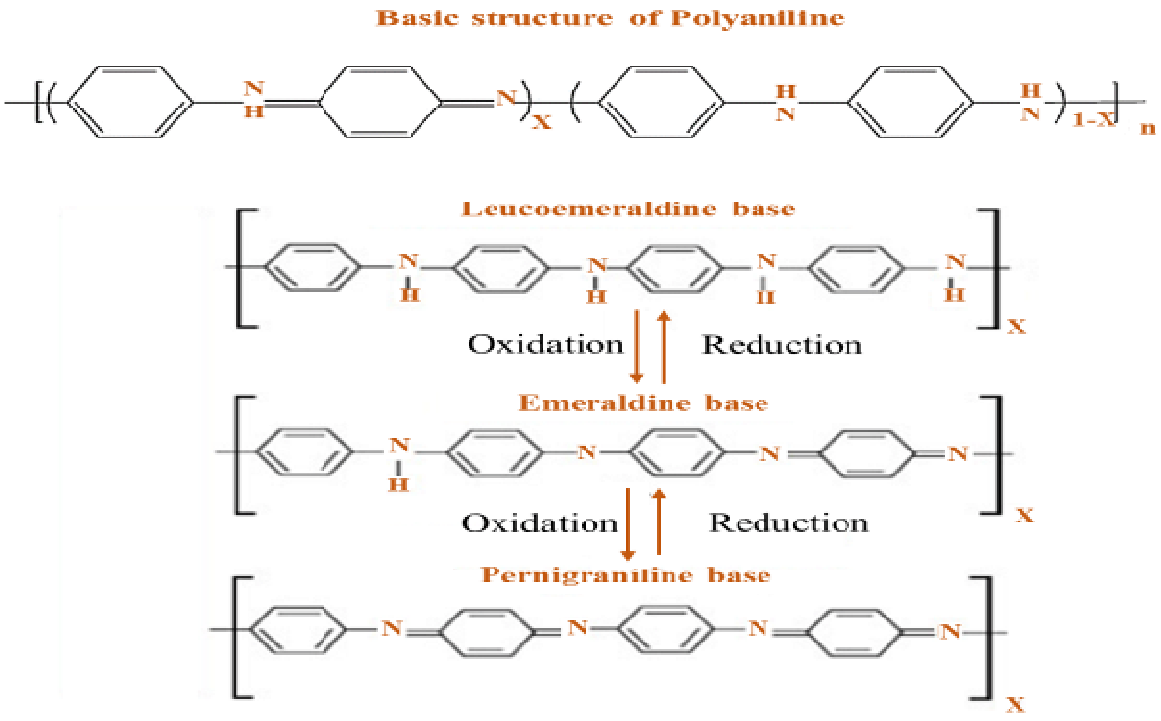
Atmospheric pressure dielectric barrier discharge (AP-DBD) was used to deposit the coatings. The discharges were produced between two plane parallel electrodes generating a 19cm^2 plasma discharge area with electrode gap 1mm, the sample placed in between. The droplet size distribution of the precursor spray was centrered around 1µm. A corona generator with a 10kHz sinusoidal signal was used to substrate plasma. Modulated electrical excitation was used to deposit coatings. The applied voltage, the current and the charge were determined using sutiable processes. The average power value was given as

$$Wa\ (W) = W_{peak}\ (W) \times t_{ON} / t_{ON} + t_{off.}$$

Functionlized Coatings From (PPP):-

Going through an other work by marisol Ji et. al. [47]. Where they used organic precursor as gaseous acetylene (Air Liquid) and liquid acrylic acid (sigma-Aldrich). These were filled in a quartz tube in a low pressure. They used elastomers as Poly (acrylonitriel butadiene) rubber (NBR) and fluoroelastomer (FKM) respectively. The polymerization process was carried out in the chamber and vaporised tanks to the low pressure.

The process of plasma polymerization was carried out in a capacitance radio frequency (RF, 13.56mHz) plasma reactor. The residual pressures between 10^{-5} to 10^{-4} mbar was maintained with working pressure around 10^{-2} mbar. Two plates are separated by 12cm and powered with a caesor RF generator and a glow discharge is injected between them. Plasma discharges were created for varying atmospheres.



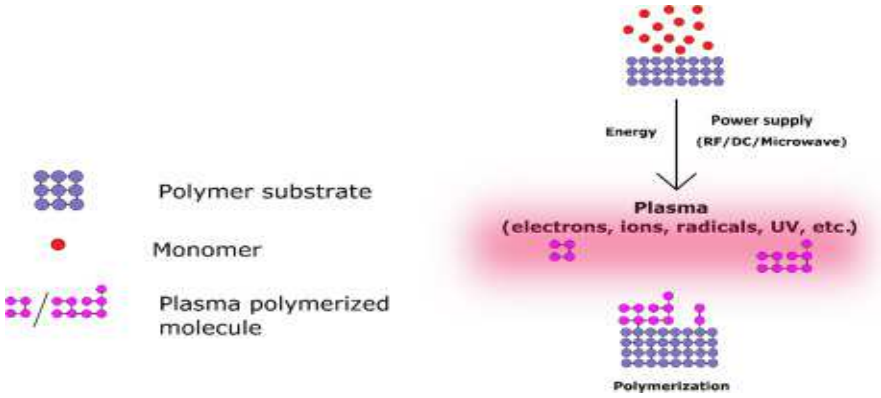


Fig.2. Plasma Polymerization

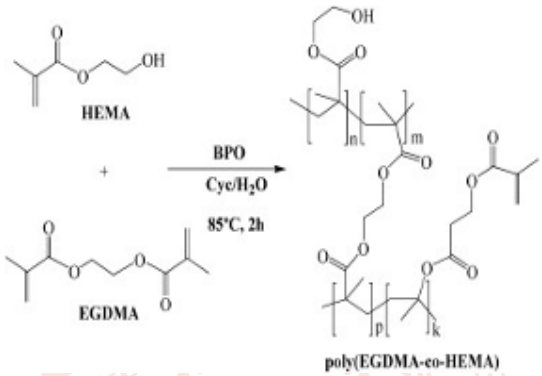


Fig.3. 2- hydroxyethyl methacrylate monomer (HEMA)

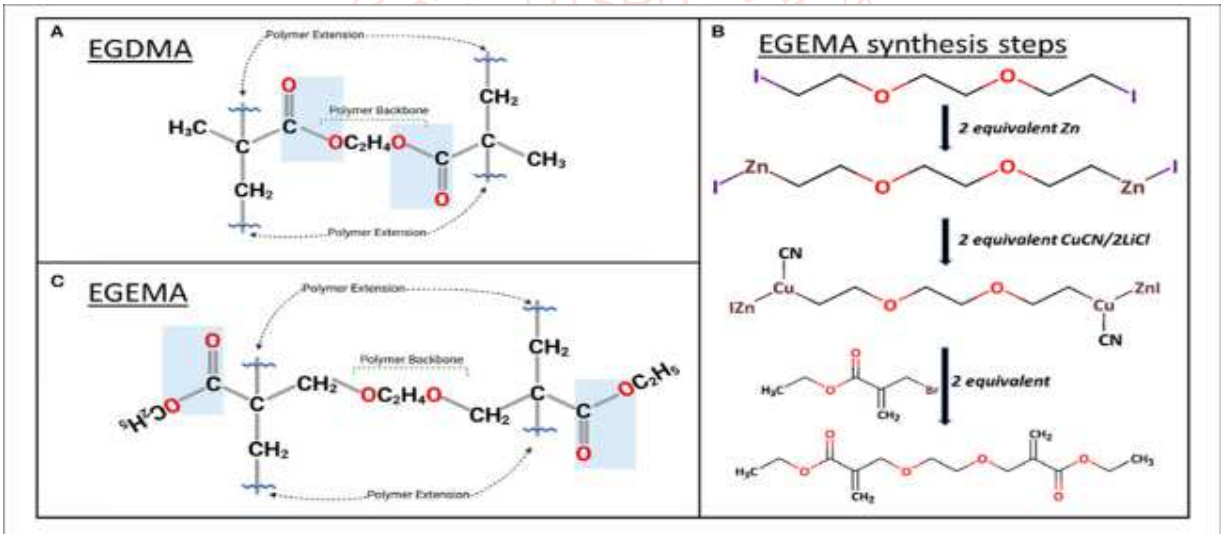


Fig.4. Molecular structure of EGDMA homo polymerization

Preparation of PANI Thin Films:-

As in the entire review and analysis we have taken polyaniline (PANI) as one of polymers. Here is brief preparation process of the same. The pulsed plasma polymerization of PANI is done in a plasma reactor by pulsing of RF plasma. In which monomer injector also operates in a pulsed monomer.

The oscilloscope is provided to control the injection of vaporized monomer. As the pulse of monomer enters the evacuated reactor, the maximum part of liquid is vaporized by flash boiling and rest is disintegrated into droplets, collected by mesh separator at 20cm form injector nozzle. When the pressure reaches nearly 40Pa the aniline pulses are injected in 10ms. Thin films are deposited with a state fill of vapour.

Fig.5. Formation of PANI thin film

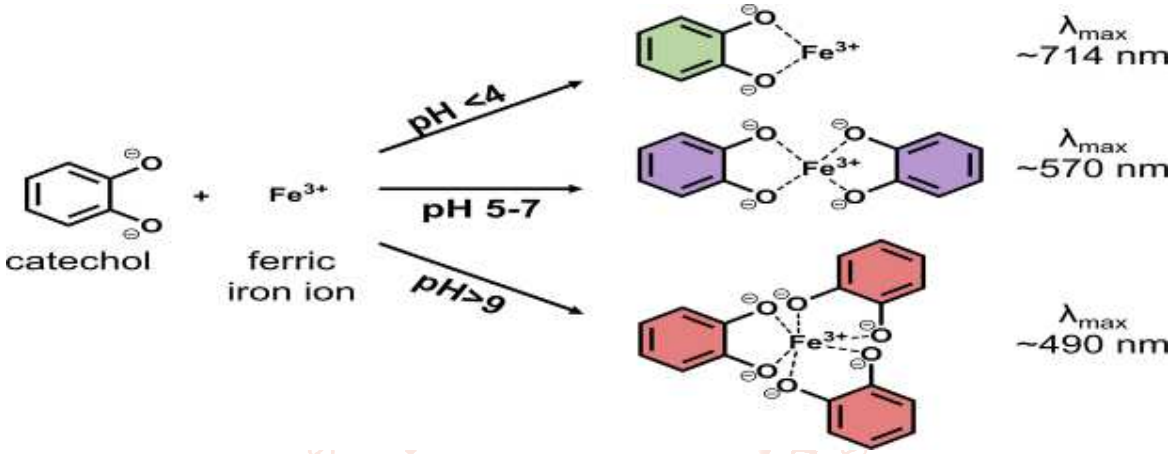


Fig.6. Formation of Catechol

Film Characterizations:

The PANI thin films prepared by inductively coupled pulsed plasma polymerization (PPP) were characterized using UV-Vis, FTIR, and other techniques like optical microscopy. The films grown on glass microscope slides were used for FTIR measurements and optical absorption spectra was carried out with dc-nulling optical transmittance bridge.

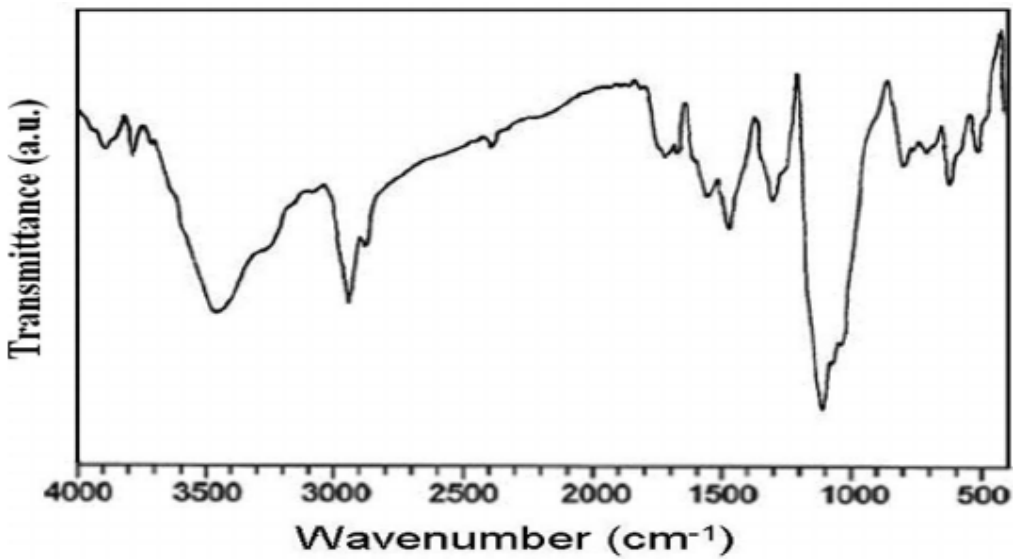
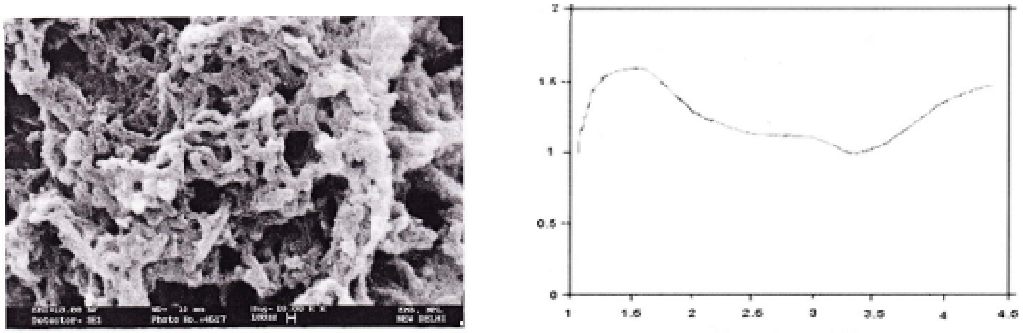


Fig.7. FTIR Spectra of unstirred synthesized polyaniine FTIR spectra of unstirred synthesized polyaniline



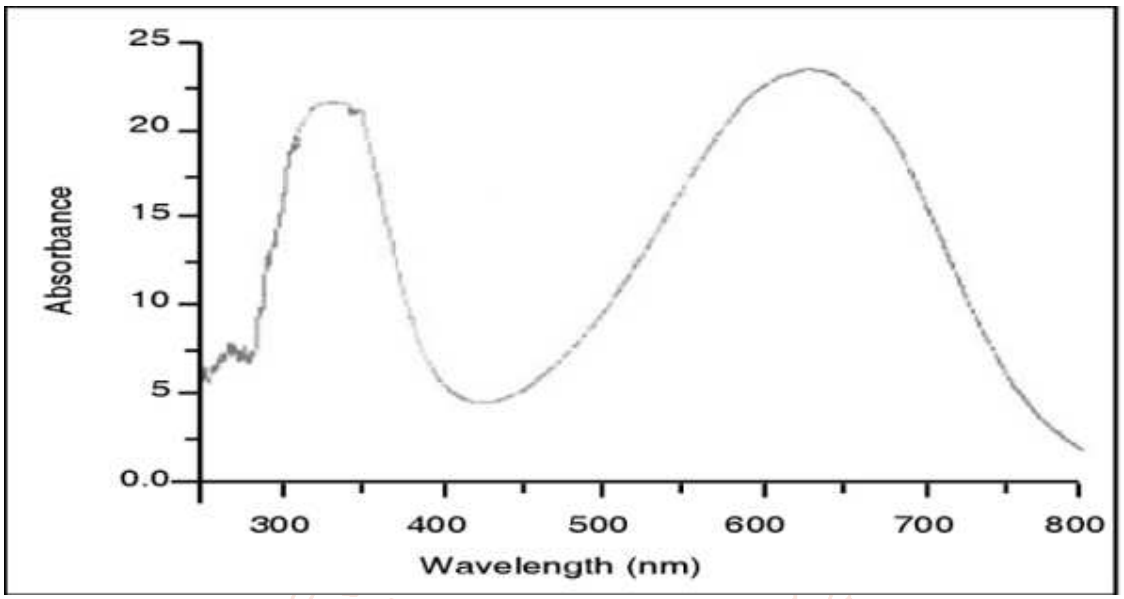


Fig.8. Optical absorption spectra as a function of the photon energy of PANI thin film

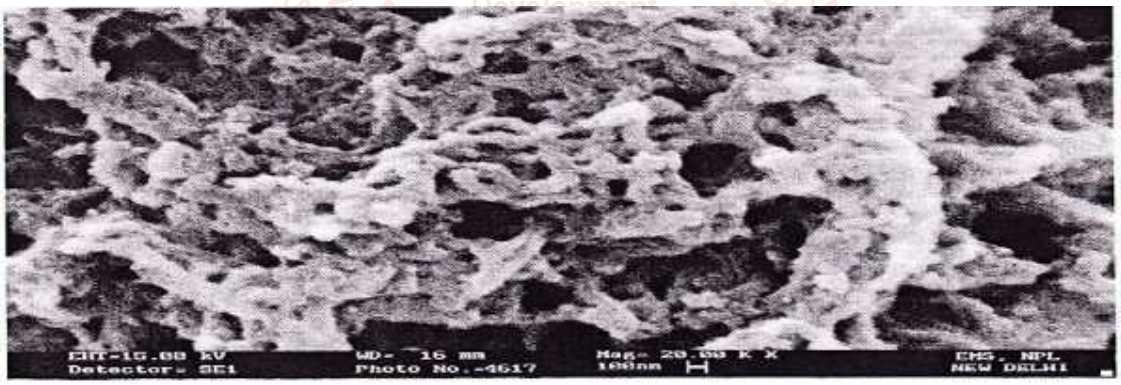


Fig.9. SEM Image of thin films

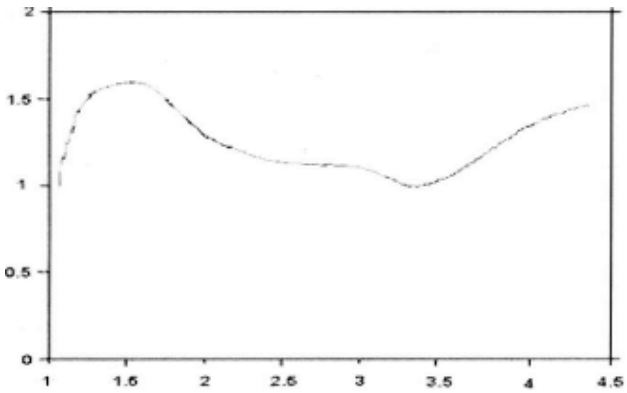


Fig.10. Optical absorption spectra as a function of the photon energy of PANI thin film

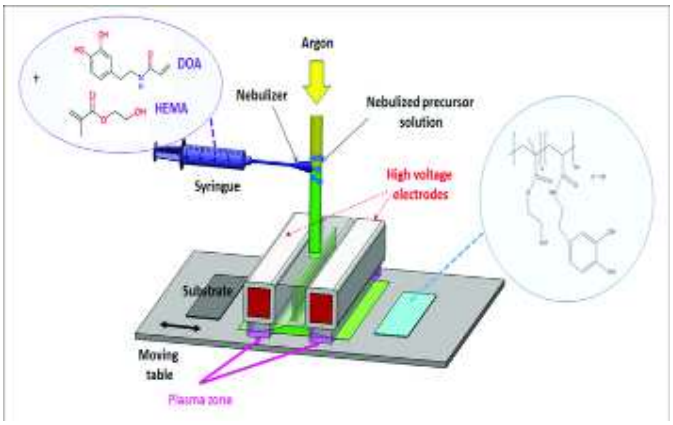


Fig.11. Schematic representation of the atmospheric aerosol assisted plasma process.

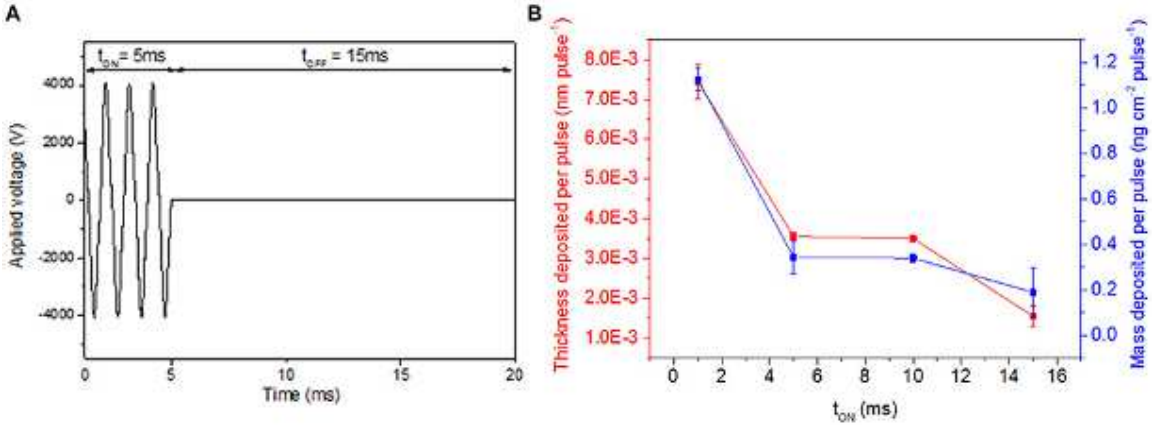


Fig.12. (A) Example of pulsed voltage with t_{ON} of 5 ms and t_{OFF} of 15 ms (B) mass deposited per pulse (i.e., t_{ON} + t_{OFF}) for a 15 ms t_{OFF} and t_{ON} values ranging from 1 to 15 ms (error bars: means ±SD, n=3.

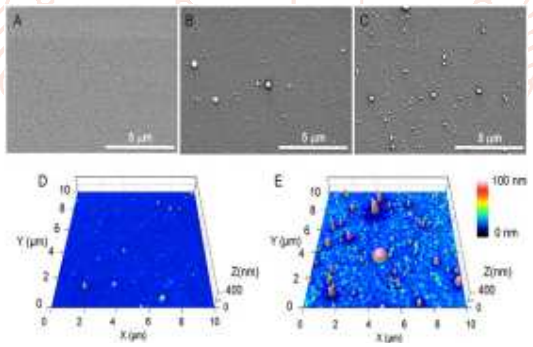


Fig. 13. SEM pictures for coatings deposited at 15 ms t_{OFF} and t_{ON} of: (A) **1 ms,** (B) **5 ms, and** (C) **10 ms. AFM pictures for coatings deposited at 15 ms t_{OFF} and t_{ON} of:** (D) **5 ms and** (E) **10 ms.**

A

XPS chemical composition, at. %					
	C	O	N		
DOA-HEMA mixture (theory)	66.99	33.00	0.01		
pp(HEMA), 1:15ms	71	29	-		
pp(DOA-HEMA), 1:15ms	71	29	-		
pp(HEMA), 10:15ms	73	25	2		
pp(DOA-HEMA), 10:15ms	74	25	1		
XPS C1s deconvolution, %					
	C-C	C-COO	C-O	C=O	COOR
pp(DOA-HEMA), 1:15ms	41	16	22.5	4.5	16
XPS C1s deconvolution, %					
	C-C	C-COO	C-O	C=O CONHR	COOR
pp(DOA-HEMA), 10:15ms	51.1	11	22.3	4.6	11

B

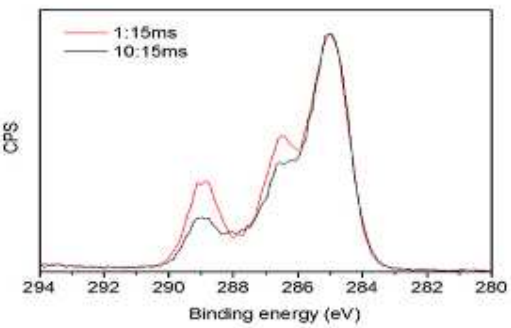


Fig.14. Table summarizing XPS analyses for pp(HEMA) and pp(DOA-HEMA) deposited at 1:15 ms and 10:15 ms (A)**, XPS C1s envelope overlaps of pp(DOA-HEMA) deposited at 1 and 15ms t_{ON} and 15 ms t_{OFF}**(B)**.**

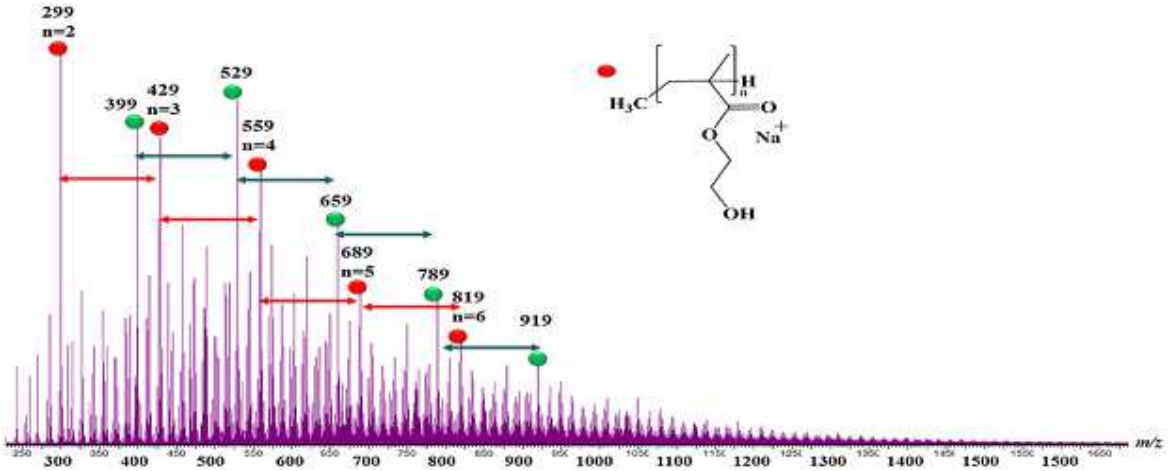


Fig.15. ESI mass spectrum recorded for a pp(DOA-HEMA) deposited in a 1:400 ms pulsed mode.

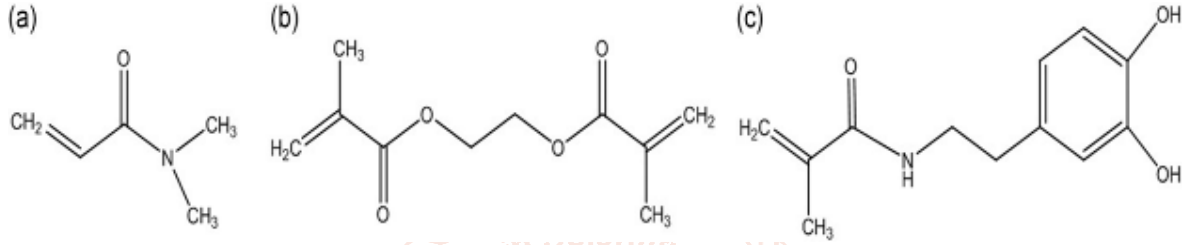


Fig.16. Chemical schemes of (a) liquid N,N-dimethylacrylamide (DMA), (b) liquid ethylene glycol dimethacrylate (EGDMA), and (c) solid dopamine methacrylamide (DOMA)

Factor	T_{off} µs	P(w)	Q(sccm)	T(mint)
T_{off} (µs)	0.06		0.2	0.01
P(w)		0.08	-0.03	-0.07
Q(sscm)		0.06	-0.06	-0.20
T(mint)				-0.21

Table 1: Effect (diagonal terms) and interaction (extra-diagonal terms) values calculated from the DoE with different factors for pp-AA.

	CC/CH response			CO/CH response		
Factor	T_{off} (µs)	P (w)	t(mint)	T_{off} (µs)	P(w)	t(mint)
T_{off} (µs)	0,36	-0,19	-0.06	-0,48	-0,15	-0,19
P(w)		-0,19	0.02		0,42	0,21
T(mint)			-0.25			0,81

Table 2: Effect (diagonal terms) and interaction (extra-diagonal terms) values calculated from the DoE with different factors for pp-AA.

S. NO.	Experimental condition	Thickness (nm)	Average deposition rate (nm/pulse)
1.	13 Pa, 50 plasma pulses	32.78	0.66
2.	13 Pa, 100 plasma pulses	98.80	0.99
3.	27Pa, 50 plasma pulses	88.64	1.77
4.	27 Pa, 100 plasma pulses	157.5	1.57
5.	40 Pa, 50 plasma pulses	236.4	4.72
6.	40 Pa, 100 plasma pulses	314.7	3.15

Table 3: Film Thickness and Average Deposition Rate of PANI Thin Film

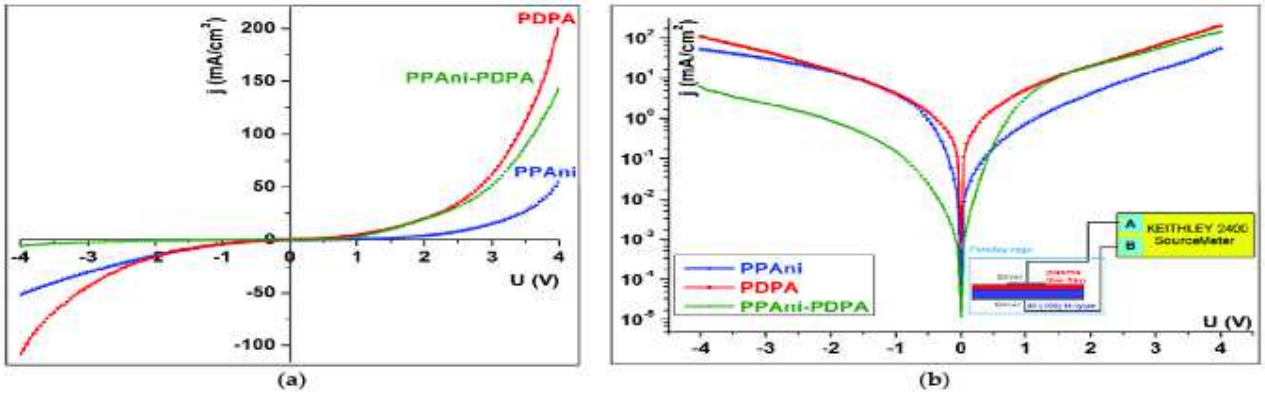


Fig.17. The plot of the current-voltage of the PPAni, PDPA, and PPAni-PDPA (a) linear plot; (b) semi-log plot.

In atmospheric aerosol assisted PPP. Ultraviolet-visable (UV-Vis) absorption spectra was taken using Perkin Elmer 950 UV-Vis NIR with 2ml resolution. FTIR was carried out in the Attenuated Total Reflectance (ATR) mode using [45] Hyperion 2000 spectrometer. X-Ray photoelectron spectroscopy (XPS) was done with Kratos Axis Ultra DLD instrument.

In functionalized coatings PPP, FTIR spectroscopy was performed with Burker Vertex 70v spectrometer in the range 4000-400cm^{-1} and resolution 2cm^{-1} after 10minutes of sample deposition. XPS was done with Kratos Axis Nova France. The Near Edge X-ray fine structure analysis-spectroscopy (NEXAFS) was performed at Helmholtz-Zentrum Berlin fur Materialien und Energie (HZB). All concerned characterization modes are shown in the diagrams.

Conclusions:-

In our discussion in summary, plasma polymerization of polymer films by pulsed plasma technique has become an advance and replicated method due to the advantage of simplified equipments, lower cost, lower thermal temperature and eco-friendly waste. In this review, we have presented different pulsed plasma polymerization techniques depending on the type and state of precursor material selected, the nature and separation of the electrodes in the chamber and the injection of monomer into the plasma reactor at desired pressure. Coatings were deposited with pulsed waves with different pulses at different voltages and under the RF plasma operated in pulsed monomer. All techniques got successful in the deposition of films.

With the advent of novel methods in the near future some methods will become ideal references for their adoption in the industry because of their potential in the synthesis of thin films. Therefore it is important to incentivize and support the research in novel methods of pulsed plasma polymerization methods of synthesis to meet the future challenges of their mass production. This will accomplish need of students, researchers and industry.

Author Contributions:-

All authors contributed equally in the conception and reviewing references. Sunil Singh. and Kajal Kushwaha wrote the draft for preparing manuscript. All authors are unanimously agreed for publications of manuscript.

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